Inventors: Villalobos et al

Serial Number:

Patent Application

Navy Case Number: 84,352

What is claimed

1. A product that is essentially devoid of a sintering aid components comprising spinel that has

porosity of less than 0.2 %, is transparent to light having wavelengths in the range of 0.4-5.5

microns, has uniform properties, is devoid of grains larger than about 1 mm and is devoid of

grains of exaggerated size.

2. The product of claim 1 wherein its spinel grains are less than about 300% of the average-

sized grain.

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3. The product of claim 1 having transparency in excess of 50% and its spinel grains are within

about 300% of the size of an average grain.

2. The product of claim 1 wherein its spinel grains are less than about 300% of the average-

sized grain.

4. The product of claim 3 having transparency of at least 60% at a wavelength of 4 microns and

the spinel is a hard crystalline solid selected from the group consisting of oxides of magnesium

and aluminum.

5. The product of claim 2 having transparency of at least 60% at a wavelength of 4.0  $\mu$ m and the

spinel is a hard crystalline solid MgAl<sub>2</sub> O<sub>4</sub>.

6. A process for preparing a transparent ceramic product comprising the steps of:

(a) dissolving a sintering aid in a suitable solvent to form a sintering aid solution,

(b) applying the sintering aid solution to ceramic particles to form a ceramic dispersion,

(c) sub-dividing the ceramic dispersion to form droplets comprising at least one ceramic

particle coated with the sintering aid solution,

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(d) drying the droplets to form dried coated particles comprising at least one ceramic particle coated with a dried layer of the sintering aid, and

- (e) densifying the dried coated particles to form a transparent ceramic product having uniform optical and mechanical properties and being devoid of grains larger than about 1 mm and grains of exaggerated size.
- 7. The process of claim 6 wherein said densifying step is carried by applying minimal pressure of about 50 psi to the coated particles while raising temperature to above the melting temperature of the sintering aid and pressing the dried coated particles at above about 5000 psi while increasing temperature to above about 1500°C.
- 8. The process of claim 6 wherein said ceramic particles are spinel MgAl<sub>2</sub>O<sub>4</sub>, wherein said densifying step is accomplished in a hot press by ramping temperature from ambient to above 1500°C.
- 9. The process of claim 6 wherein said ceramic particles are spinel MgAl<sub>2</sub>O<sub>4</sub>, and wherein said densifying step is accomplished by ramping steps to an elevated temperature with intermittent holding periods to allow the sintering aid to liquify and escape.
- 10. The process of claim 9 wherein the ramping steps are about 20°C/minute and the holding periods are between the ramping steps and are about one half hour.
- 11. The process of claim 10 wherein there is an initial ramping step to a temperature of less than 100°C above the melting point of the sintering aid followed by a holding period to liquify the sintering aid, an intermediate step to less than about 550°C above the melting point of the sintering aid, followed by a holding period to allow vaporized sintering aid or components

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thereof to escape, and a final ramping step to above 1500°C, followed by a holding period to fully densify the dried coated particles to a transparent spinel product.

- 12. The process of claim 8 wherein the spinel particles making the spinel dispersion have particle size in the range of 500nm to 100  $\mu$ m; wherein the solvent includes water and an additive selected from the group consisting of ethanol, isopropanol, and mixtures thereof; and the ratio of water to hydrocarbon to LiF sintering aid to spinel particles is about 220 ml, about 780 ml, 0.2 grams, and 10 grams, respectively.
- 13. The process of claim 12 wherein pH of the final sintering aid solution is about 7

  14. A process for preparing a transparent MgAl<sub>2</sub> O<sub>4</sub> spinel product having maximum transparency in excess of about 60% comprising the steps of:
- (a) dissolving LiF sintering aid in water to form a sintering aid solution of about a neutral pH,
- (b) mixing the sintering aid solution and a volatile, low surface tension/viscosity additive selected from the group consisting of ethanol, isopropanol, and mixtures thereof,
- (c) applying the sintering aid solution to MgAl<sub>2</sub> O<sub>4</sub> spinel particles to form a spinel dispersion,
- (d) atomizing the spinel dispersion to form droplets comprising at least one spinel particle coated with the final spinel solution,
- (e) drying the droplets to form dried coated particles comprising at least one spinel particle coated with a dried layer of the sintering aid, and
  - (f) densifying the dried coated particles to form a transparent MgAl<sub>2</sub> O<sub>4</sub> spinel product

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having uniform optical and mechanical properties in absence of grains of exaggerated size.

15. The process of claim 14 wherein said densifying step is accomplished in a hot press by

ramping temperature from ambient to above about 1500°C.

16. The process of claim 15 wherein said densifying step is accomplished by ramping steps to

an elevated temperature with intermittent holding periods to allow the sintering aid to liquify and

escape.

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17. The process of claim 17 wherein the ramping steps are about 20°C/minute and the holding

periods are between the ramping steps and are about one half hour.

18. The process of claim 17 wherein there is an initial ramping step to a temperature of about

950°C followed by a holding period to liquify the sintering aid, an intermediate ramping step to

about 1200°C, followed by a holding period to allow vaporized sintering aid to escape, and a

final ramping step to above 1500°C, followed by a holding period to fully densify the dried

coated particles to a transparent spinel product.

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